A Quartz - Crystal Film - Thickness Monitor

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By
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CERTIFICATE



Certified that this work has been carried out under our supervision and that this has not been submitted elsewhere for a degree.

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This thesis has been approved for the award of the Degree of Master of Perina agy (M. Tech.) in accordants with the regulations of the Indian Institute of Technology Kanpur Dated. 22/1.

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LIST OF SYMBOLS

A	Area	of	the	quartz	crystal	or	gain	of	amplifier
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Area of fim deposited on quartz crystal

Ar Gain for ramp input

A_v Voltage gain of amplifier

B, B D-C power supplies

C Shear constant for the quartz crystal

d Shift in the fringes

D Distance between successive fringes

f Frequency of the quartz crystal oscillator

f Natural frequency of the quartz crystal

m Mass of film deposited on the crystal

t Thickness of the crystal

tf Thickness of the film

B Feedback factor

P Density of the crystal

Pf Density of the deposited film

ABBREVIATIONS

A = Angstrom unit = 10⁻⁸ cm

 $\triangle f = Change in frequency$

K = Kilochms

M = Magachms

Micron= 10000Å or 10⁻³ torr

Torr = Pressure corresponding to 1 mm of mercurry column

Synopsis

Thin films of metals, dielectrics and semiconductors are extensively used in the fabrication of electronic devices. For example thin film resistors and capacitors are used in hybrid circuits. Metallisation forms an important process step in the manufacture of Integrated Circuits. The film thickness as well as the rate of formation of the film play important roles in controlling the performance of these devices. Thus, the measurement and control of these parameters while the film is being formed becomes essential.

The thesis deals with the design and fabrication of thickness and rate monitor based on the quartz crystal oscillator technique. This method combines simplicity and high sensitivity for mass measurements. The principle consists in measuring the frequency change of an oscillator caused by the deposition of mass on a quartz crystal which controls the oscillator frequency.

The monitor oscillator circuit must yield sustained oscillations for a number of similar crystals and during deposition. Further the widely observed mode jumping phenomenon must be eliminated. A solution to these problems is achieved by incorporating automatic loop gain control in the oscillator circuit. Reliable operation of the circuit is obtained by gold plating the crystal faces.

The change in the frequency of the oscillator is measured using a digital frequency counter. A system consisting of a local

oscillator, mixer, low pass clipper amplifier, monostable multivibrator averaging and differentiating amplifiers produces analog outputs proportional to film thickness as well as rate of deposition. The system is designed using integrated circuits. The design details of the system are presented.

A frequency stability of 1Hz per hour has been obtained with water cooled monitor oscillator. A mass measurement sensitivity of the order of 10⁻⁹ gm/cm² has been achieved. The system has been calibrated for thickness measurements of silver and copper films.

CHAPTER 1

INTRODUCTION

1.1 General discussion on thin films:

It is over a century that thin films are being used for many applications and tremendous amount of investigation is going on to understand the basic properties of thin films of various materials. Thin films were first employed as coatings of precious metals for protection against corrosion. The rapid growth of thin film technology started between the two world-ward when vacuum evaporated metal and dielectric films were used in optics for mirrors and antireflection coatings respectively. But it was their potential usefulness for the electronic industry which stirred scientists to probe deeply into the properties of thin films. At present thin films find applications in almost all fields of science and technology.

The study of optical properties has led to a specialized technology of "optical interference films". Study of electrical properties of metals and semiconductors has led to miniaturized, more reliable components, devices and circuits. Magnetic properties are widely used for making computer memory. Thin films have become a basic tool of investigations in metallurgy and in study of mechanical properties, electron scattering, surface properties of

semiconductors, growth of nuclei etc.

1.2 Film thickness and its effects on basic properties:

The term 'thin film' widely used in todays science and technology, can be defined as a layer of gaseous, liquid or solid matter. Films are considered thin in the general sense when one dimension (i.e. thickness) is much smaller than the other two dimensions. 'Thickness' has a meaning only when a film is continuous and uniform but in ultra-thin films ranging from a few angstroms to several tens of angstrom the film is discontinuous and consists of islands. Therefore here we talk of "average thickness".

Film thickness is a very important parameter for basic investigations as well as for film applications. This is understandable, for the very fact that one dimension (thickness) is much smaller than the other two gives rise to properties which differ in the film form from those of the bulk material.

Consider a thin film of resistive material. Not only will the resistance of the film increase with decreasing thickness because of the reduction in cross-sectional area, but also additional effects appear as the mean free path of the conduction electrons becomes comparable or even greater than the film thickness. Also, in ultra-thin films the conduction mechanism itself changes (Neuge - bauer and Webb 1962).

In thin film devices such as capacitors and insulated gate FETs the dielectric determines the electronic performance. The significant properties are the conductivity and breakdown voltage and their variation as a function of thickness.

In magnetic film devices, the film thickness has a strong influence on the demagnetising and reverse magnetising field and on the domain wall structure.

In thin film optics the thickness affects the refractive index, transmittance and reflectance. Almost all applications in thin film optics require coatings of well defined and precisely controlled thickness. For example, in interference filters, where a bandwidth of 10 Å is to be achieved, the error in film thickness should be less than 0.6%.

The thickness of thin films of superconducting material plays an important role in determining the transition temperature and critical magnetic field.

1.3 Thin film deposition processes:

The various deposition techniques (Holland 1965; Hall,
Berry and Harris 1968) are electrodeposition, vapour deposition,
cathode sputtering and vacuum evaporation. Electrodeposition is
mainly used for depositing noble metal films on other metals to

prevent corrosion. For electronic application this has little use. Vapour deposition is widely employed for growing single crystal epitaxial layers specially in silicon planar technology. Cathode sputtering is widely used for depositing refractory metals or high melting point oxides. Vacuum evaporation is done at pressures lower than 10⁻⁵ torr and the evaporation is done either by a heated filament (or boat) of a refractory metal or by an electron bombardment arrangement.

A quartz crystal monitor is used widely with the vacuum evaporation technique and it can also be used with cathode sputtering technique. In the present work, vacuum evaporation by heated filaments of tungsten, molybdenum or tantalum is used for depositing the films.

1.4 Techniques of measuring film thickness:

Methods of thickness determination have been reviewed by many workers. (Behrndt 1966, Gillespie 1967 and W. Steckelmacher 196

Conventional ways of measuring thickness fail when
the film thickness is of the order of angstroms. Only "Optical
interference" methods measure absolute thickness while all other
methods measure some other parameter which depends on the thickness
in a known manner. Alternatively, film thickness can be calculated
from rate and time of deposition. Thus the methods, either measure

thickness directly or measure the rate directly. A knowledge of deposition rate is always desirable, as film structure and properties are dependent on the rate.

The methods used in monitoring thickness are:

- i. Rate monitoring: Rate meters give both rate and thickness. Rate meters can be employed for all materials. Ionization gauge rate meter, rate meter employing movement of current meters and rate meter based on the momentum of arriving atoms are the ones which are widely used in this category.
- ii. Thickness monitoring: Optical methods, electrical methods, gravimetry and quartz crystal oscillator come under this category.

The change in the frequency of a quartz crystal is dependent upon the deposited mass. Thus, if a crystal is placed so that material is deposited upon it, (at the same rate as upon the substrate) the frequency change in the crystal (oscillator frequency) will be an accurate measure of the average thickness. While the thickness monitor based on this principle is described in detail, the principle of Multiple Beam Interferometer is briefly discussed in the Appendix A.

1.5 Aim of work:

The aim of work has been to design and fabricate a versatile

system based on the quartz crystal oscillator for measuring thickness of thin films of materials deposited either by evaporation or sputtering.

Since the sensitivity and accuracy of thickness measurement to is limited by the inherent frequency instability of the crystal oscillator, effort has been made to construct a crystal holder and an oscillator circuit which can give a frequency stability of about 1 Hz in 6.5 MHz over an interval of one hour. In other words the accuracy of thickness determination comes to a fraction of Angstrom for most materials.

When the crystal is loaded with a thin film of the material, the frequency of the oscillator reduces. With large reductions the crystal tends to oscillate at an overtone frequency. This phenomenon is known as "Mode Jumping". The undesirable mode jumping was effectively avoided by keeping the loop gain in the oscillator just above unity by employing automatic gain control.

Analog indications of thickness and rate of deposition have been provided. The quartz crystal monitor is designed using integrated circuits. The design of a compatible "automatic rate control unit" is outlined in chapter 7.

The Quartz crystal monitor has been calibrated for two materials

namely copper and silver.

A Multiple Beam Interferometer has been assembled which was used to calibrate the Quartz crystal monitor.

QUARTZ CRYSTAL OSCILLATOR AS A TRANSDUCER

2.1 Quartz crystal microbalance:

Quartz crystal oscillators and narrow band crystal filters have been used widely. (Mason 1950, Heising 1946). Quartz crystal acts as an electromechanical resonator of high Q and hence yields a precise frequency.

In the present work quartz crystal oscillator is used as a transducer. Mass loading on the crystal faces reduces the frequency of the crystal oscillator and this reduction is a measure of the mass deposited. Quartz crystal oscillator shows extreme sensitivity to mass loading. For vacuum evaporations, the sensitivity of the crystal oscillator is usually about 10⁻⁹ gm/cm². With precise temperature control, sensitivities of the order of 10⁻¹² gm/cm² have been achieved. (Stockbridge and Warner 1963). A quartz crystal microbalance combines high sensitivity with ease of fabrication and operation. Monitoring both thickness and rate, automatically, is also possible.

2.2 Monitor crystal selection:

Quartz crystal can have various modes of vibration (Mason 1950, Cady 1964). The thickness shear mode is the only one of interest in this particular application. In thickness shear mode an antinode is formed on the faces of the quartz crystal plate and only the mass of the material deposited affects the frequency (Figure 1a). Y, AT

and BT cut crystals in the form of rectangular or circular thin plates are used for thickness shear mode. The major criterion in choosing a particular crystal cut is the temperature co-efficient of frequency. Figure 1b shows the temperature co-efficient of frequency versus the rotation about x-axis (Mason 1950). At a given temperature BT and AT cuts are those which show a zero temperature co-efficient of frequency. Figure 1c shows the change in frequency versus temperature for AT and BT cuts. These curves show that with an AT-cut crystal a larger temperature range (with a small temperature co-efficient) can be obtained than with a BT-cut crystal. Therefore AT-cut crystals are chosen for monitor-crystals.

2.21 Theoretical relation between mass loading and frequency shift:

The frequency f_n of resonance of the AT and BT cut crystals for thickness shear mode of vibration (Mason 1950) is given by

$$f_n = \frac{N}{t}$$
 eqn. (1)

Where $N = a \text{ constant} = \frac{1}{2} \left(\frac{C}{\rho}\right)^{\frac{1}{2}}$

Where C is the shear elastic constant

P is the density of the crystal

and t is the thickness of the crystal

The value of N is (due to Heising 1946)

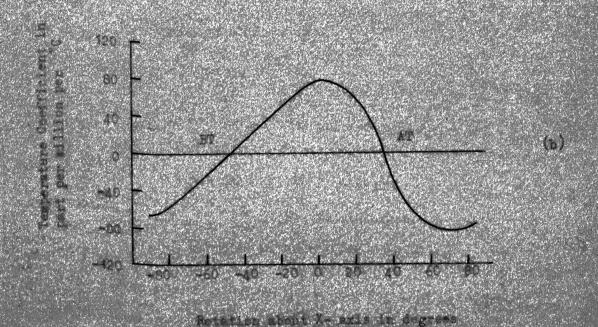
N = 1670 KHz-mm for AT cut eqn. (2

= 2500 KHz-mm for BT cut

(a)



Sectional view



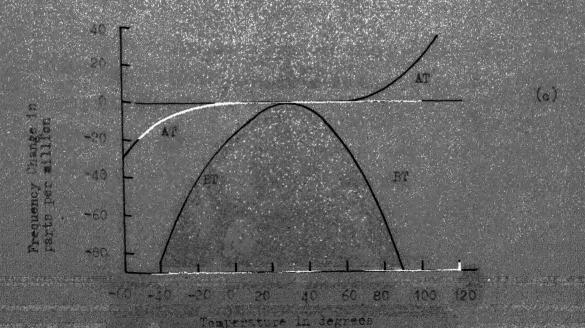


Figure: 1 CHYSTAL CHARACTERISTICS FOR THIORNESS SHEAR MODE

The relationship between the frequency change of and the mass deposited m_f has been derived (Stockbridge and Warner 1962) using the perturbation technique. Of is given by

$$\triangle f = -\frac{f_n^2 K}{NR} \times \frac{m_f}{A} = \dots \qquad eqn. (3)$$
for $\triangle f < < f_n$

and
$$\frac{f_n^2 K}{NP}$$
 = a constant = $c_f \cdots eqn.(4)$

Where K is a constant near unity depending on the distribution of mass over the crystal surface,

 m_f is the mass of the film deposited,

A is the area of the crystal face,

and Cf is the mass determination sensitivity

From eqn. (3) and eqn(4) we can define a few quantities which are useful in deciding the frequency of the crystal for a given application.

f is defined as the "mass determination sensitivity" and it incorporates all the constants of the particular crystal. For a given Δf , the larger the C_f , the smaller the mass which can be determined. Another fact to be noted is that C_f varies as the square of the natural frequency, f_n . Thus higher mass determination sensitivity is achieved with larger f_n .

The linear dependence of Δf and m_f does not hold indefinitely. If a deviation of 1% from linearity is permissible,

then \triangle f should not exceed 0.5% of f_n (Behrndt 1966). This limit of \triangle f thus decides the "maximum detectable mass, f_n ". Substitution in equation (3) gives

$$\frac{m_{\text{fmax}}}{A} = \frac{.005 \text{ N} \text{ p}}{f_{\text{n}} \text{ k}} \text{ gm/cm}^2 \dots \text{ eqn.}(5)$$

or

$$_{\text{fmax}}^{\text{m}} \propto \frac{1}{f_{\text{n}}}$$

Thus the larger the $\mathbf{f}_n,$ the smaller the "maximum detectable mass \mathbf{m}_{fmax} ".

The "smallest detectable mass m_{fmin} " is defined as the minimum mass which gives rise to the minimum detectable frequency change, Δf_{min} . Rearranging equation (3) we get,

$$\frac{m_{fmin}}{A} = -\frac{\Delta_{fmin}}{f^{\frac{2}{n}}} \times \frac{NR}{K} \text{ gm/cm}^{2} \dots \text{ eqn.}(6)$$

$$|^{m}_{fmin}| \propto |\Delta f_{min}|$$

$$|^{m}_{fmin}| \propto \frac{1}{f_{n}^{2}}$$

Another important factor is the thickness of the crystal itself. From equation (2) it becomes clear that for higher frequencies crystal must be thin and too thin a crystal requires very careful handling.

2.22 Criteria for selecting crystal oscillator frequency, fn:

From equations (2) to (6) we note that a large f_n leads to a large C_f , small m_{fmax} and small m_{fmin} . Thus a large mass determination sensitivity is obtainable for a given $\triangle f_{min}$ but then m_{fmax} is smaller. Such a condition is suitable for the study of nucleation properties of ultra-thin films. A large sensitivity is required but total mass deposited is small in itself. Typically $m_{fmin} \sim 10^{-10}$ gm/cm² and $m_{fmax} \sim 10^{-6}$ gm/cm² are desirable in such an application. Substituting these in equations (5) and (6) gives us f_n of the order of 200 MHz (assuming $\triangle f_{min} = 1 \text{ Hz}$). Such high frequencies are obtained only by exciting an overtone because for exciting fundamental the crystal becomes too thin. The frequency being high and overtone excitation being required the circuit becomes complex.

In electronic applications where desired thickness range is roughly from 100 Å to 10,000 Å, we require m_{fmin} of the order of 10^{-7} gm/cm² and m_{fmax} of the order of 10^{-3} gm/cm². Substituting in equation (6) we get f_n approximately 6 MHz (assuming $\Delta f_{min} = 1$ Hz) and m_{fmax} of the order of a few milligram. The thickness of a 6 MHz crystal (at fundamental frequency) is obtained from eqns.(1) and (2) and is approximately .3 mm which can be handled with ease. Therefore 6.5 MHz AT cut-crystals are chosen.

2.3 Frequency stability and controlling factors:

The smallest detectable mass m_{fmin} depends directly on the minimum detectable frequency change \triangle f_{min}. \triangle f_{min} is determined by the drift in the crystal oscillator frequency. By reducing the drift in the oscillator frequency we can detect a smaller mass deposited on the crystal. As \triangle f_{min} was assumed to be 1 Hz when frequency of the crystal was decided, the drift in the frequency of the oscillator should be less than 1 Hz/hour. This frequency must be determined to a compatible precision. The Hewlett Packard 10 MHz counter with least significant digit of .1 Mz is found to be adequate.

In addition to mass changes, there are several other factors which will result in frequency shift of the crystal-oscillator. Some factors control the natural frequency of the crystal itself while others control the oscillator frequency.

Variations in surrounding gas pressure, adsorption of gases etc. or moisture on the faces of the crystal change the natural frequency of the crystal (An increase of about 40 Hz was observed when chamber was evacuated from atmospheric pressure to 10⁻⁵ torr). Care must be taken to avoid mechanical shocks to the crystal which tend to change the frequency abruptly. Temperature changes cause change in crystal frequency and hence crystal must be held in a water-cooled holder.

The factors which cause drift in the oscillator frequency are changes in parasitic lead inductances and capacitances, variations in supply voltages, aging of active device parameters and changes in tuning elements (if used) due to variation in temperature.

The design of the crystal holder and the oscillator circuit due has been such as to minimise these variations, to various factors so that desired stability within 1 Hz/hour is obtained.

2.4 Design of sensor head:

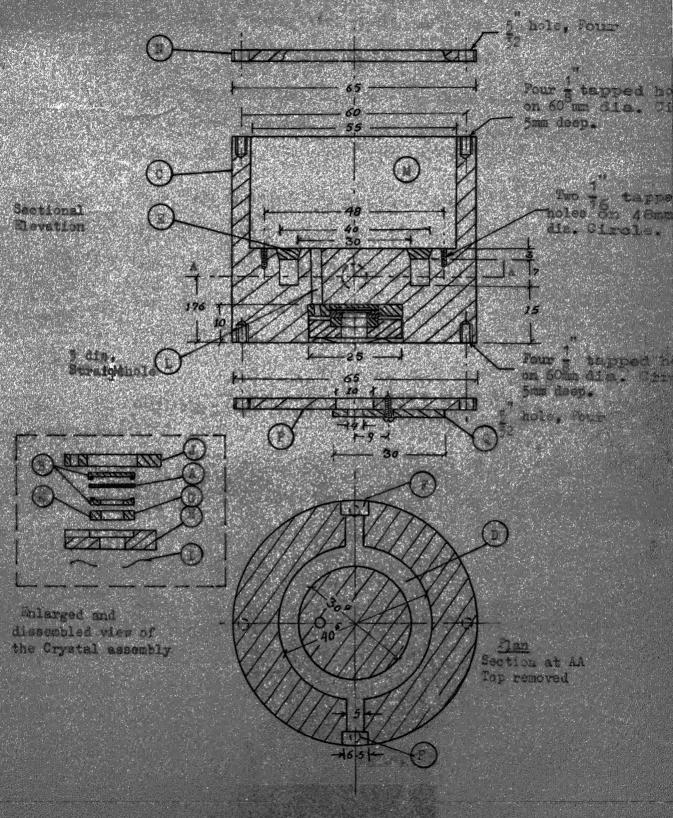
The crystal is held in a holder so as to prevent it from any mechanical shock and physical damage and to provide the electrical contacts to the electrodes on the two faces. The main object of the crystal holder design is to minimise the drifting of the crystal frequency. Here variation in the crystal temperature plays the most important part. Since the radiation from the evaporation source and also the deposition of the film heat the crystal surface, the crystal frequency drifts widely unless the crystal faces are cooled. A water cooled crystal holder has, therefore, been designed.

It was also observed that because of the long leads the frequency used to drift as much as a few hundred Hz. So to minimise the changes in the lead capacitance and inductances, a short piece of shielded cable was used and a miniaturized oscillator circuit

was installed right <u>in</u> the crystal holder. Inclusion of the oscillator circuit makes the nomenclature. "Sensor-head" more meaningful than "crystal holder". Details of the sensor head are shown in figure 2. A brief description is given below.

The crystal A is held in close contact with the holder plates B and a water-cooled stainless-steel (ss) block C. circulate water, a circular groove D is cut :n the SS block and is covered from top by welding a SS ring E. Two holes F are drilled into the sides of the SS block so that copper tubing may be welded and circulation of water become possible. The SS block makes a contact to one electrode of the crystal and cools the crystal, simultaneously providing an electrical connection to the electrode. The contact to the other electrode is made through a copper annular ring G which is isolated from the main block by a teflon disc H. Spring I provides the required pressure on the crystal. A teflon disc J, with a rectangular hole for the crystal, is used to keep the crystal in proper position. A short length of shielded cable K is soldered to the copper annular ring G at one end. The cable passes through a hole L into the chamber M which holds the oscillator circuit. The other end of the cable is connected to the oscillator circuit. The chamber M is covered with a SS plate N having insulated terminals for the power supply leads and the output lead. Another plate P is used at the crystal side to press the spring to the required pressure

Scale: Full size All dimensions in mo



Flaura!

and to provide a circular opening for the deposition on the crystal. The plate Q, having heles of different diameters, provides adjustable area of film deposited on the crystal.

Apart from minimising temperature variation, shocks and variation in the lead parasitics, the sensor-head has following additional features.

Since a stainless steel block is used with copper tubes brazed on it, the sensor-head can be safely used in a ultra-high vacuum system.

Since the crystal is pressure sandwitched between holder plates, both plated and non-plated crystals can be used for monitoring.

Oscillator design is discussed in the next chapter.

CHAPTER 3

OSCILLATOR DESIGN

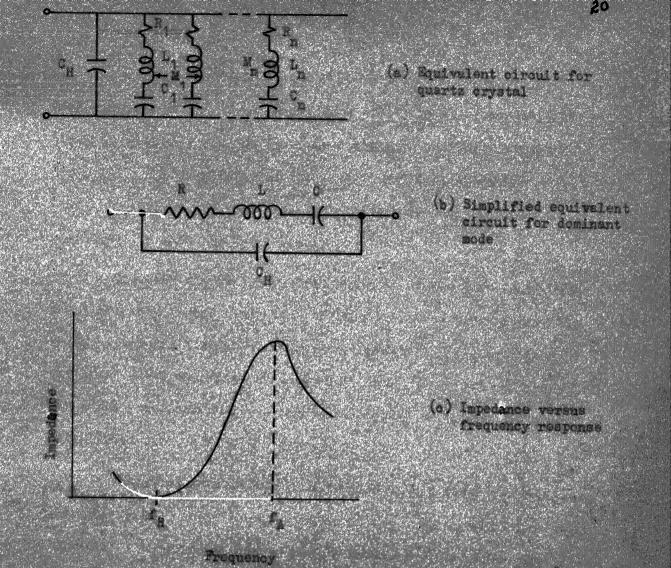
3.1 Series and parallel excited crystal oscillators:

Quartz crystal oscillators are widely used for their excellent frequency stability. In the analysis of the performance of a crystal for its use in oscillator, it is very convenient to obtain an equivalent circuit in terms of inductances (L), capacitances (C) and resistances (R). With reference to Figure 3a, the coupling between the various modes can be represented by mutual inductances (M).

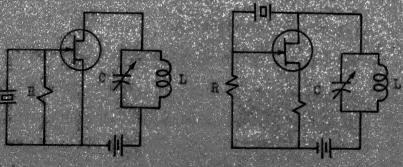
However, when there is a dominant mode, the equivalent circuit can be simplified as shown in Figure 3b. From the simplified equivalent circuit we can see that the impedance of the crystal varies as is shown in Figure 3c.

At resonance, impedance is very small (equal to R) while at anti-resonance it is very large. This property can be used to make parallel excited crystal oscillators and series excited crystal oscillators. Typical circuits are shown in Figure 3d and 3e.

Consider the Miller Circuit given in Figure 3d. Under resonating condition, the plate load is arranged to be inductive which requires that the crystal be capacitive in order that the loop phase shift be zero. Thus the frequency of oscillation will be between f_R and f_A .

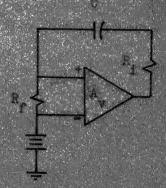


rrequency



(d) Willer oscillator (parallel excitation)

(e) Pierce oscillator (series excitation)



(f) Series excited positive feedback orystal oscillator

Figure: 3 QUARTZ CRYSTAL EQUIVALENT CIRCUIT AND OSCILLATORS

Since changes in the input capacitance will vary the frequency, the parallel resonance configuration does not give the best frequency stability. The simplicity of the circuit makes it attractive for a wide variety of applications. Colpittand Clapp circuits are variations of this configuration.

Consider the Pierce Circuit shown in Figure 3e. Again, when the plate load is made inductive, series resonance becomes possible when the crystal acts as a capacitive impedence. This condition is satisfied for frequencies slightly less than f_R . Since the crystal has small impedence near f_R , the shunt impedences across the crystal have little influence on the frequency.

The tuned circuit can be dispensed when a two stage amplifier providing a 360° shift between its input and output is employed (figure 3f). Crystal is then excited to series resonance. The frequency of the oscillator is f_R since crystal must be resistive to satisfy the loop phase condition. Such configuration improves the frequency stability. Much better frequency stability is achieved by using amplitude stabilization and well regulated power supplies. Meacham Bridge configuration (Meacham 1938) has the best frequency stability of all the existing crystal oscillator circuits.

Looking to the equivalent circuit of the crystal (Figure 3a), we find that the impedence becomes small (equal to $R_{\rm n}$) at a number of

discrete frequencies. Thus for series resonance, the loop phase condition (with reference Figure 3f), is satisfied at a number of frequencies and this creates a problem called as Mode Jumping. This problem is discussed in the next section.

3.2 Mode jumping problem:

It is observed that a crystal oscillating in fundamental mode jumps to a higher mode when one tries to deposit a film of large thickness. As the deposition continues modes other than thickness shear mode, get favourable condition and thus the oscillation in thickness shear mode comes to a stop. Thus the problem is to keep the crystal oscillating in the fundamental mode all the time. Once the crystal jumps mode (or stops oscillating), the information about the thickness and rate of deposition is lost and the instrumentation becomes useless.

3.21 Analysis:

A crystal has, for its electrical equivalent circuit, a set of series R,L,C circuits shunted by the capacitance between the electrodes. In the equivalent circuit coupling between these modes is indicated by mutual inductances. The coupling between various modes depends largely on the workmanship of the crystal faces and edges; and to a lesser extent on the nonuniformity and eccentricity of the deposited film. These dependances are very complex in nature. In the ideal case of infinite parallel flat faced disc, the analysis

of acoustic vibrations show that crystal has least losses in the fundamental mode and so on. So we can write

$$R_1 < R_3 < R_5$$
 etc.

(subscript 1,3,5 are used for the odd harmonics of the fundamental) So the equivalent circuit can be represented by simple resistances R_1 , R_5 , R_5 etc. at the frequencies f_1 , f_3 , f_5 etc.

With series resonant circuit as shown in Figure 3f, we have A_v real and positive gain. Therefore to satisfy $A\beta=1$ the condition for oscillation, β the feedback factor must also be real and positive. This is possible only at frequencies f_1 , f_3 etc.

Now $oldsymbol{eta}_{n}$, the feedback factor for n th harmonic is given by

$$\beta_n = \frac{R_f}{R_i + R_f + R_n}$$

Since for the ideal case $R_n > R_{n-1}$, therefore

That is β_1 is greater than β_3 , β_5 etc.

Now choosing $A_{\nu} \beta_{1} \gtrsim 1$ (generally known as "overdrive" in oscillator design), we make sure that $A \beta \lesssim 1$ for n=3, 5, 7 etc. Thus only the fundamental mode causes oscillations. But if sufficient overdrive is provided,

$$A\beta_n > 1$$
 for $n=1,3,2L+1$ $A\beta_n < 1$ for $n=2L+3,2L+5$, etc. where $L_{s=0}$, or 1 or 2 etc.

Larger overdrive tends to make condition $A\beta=1$ favourable for higher modes as well. There is a tendency to keep oscillations at that frequency for which $A\beta$ is greater than and nearest to 1. (Edson 1953).

In the non-ideal case, the various modes have coupling between each other and thus they add to the ideal response wherein only one mode was assumed to be in operation. For example, if 5th harmonic has a large coupling with the fundamental then eventhough the oscillations are still at f₁, a large distortion due to 5th harmonic is also present. This contribution of the higher harmonics to the fundamental is largely determined by Workmanship of the crystal faces, Overdrive and deposition of film.

Experimental results from a study of the influence these factors on the oscillator waveform are shown in Figure 12. To have self-sustained oscillations for a number of crystals some overdrive is essential. Overdrive is also essential to maintain oscillation inspite of variations in circuit parameters due to aging and variations in supply voltages.

From Figure 122 we find that the larger the overdrive, the larger the harmonic content in the oscillator output waveform. With further increase in overdrive the crystal jumps mode (Figure 12b). Thus we need an oscillator circuit which has an initial overdrive,

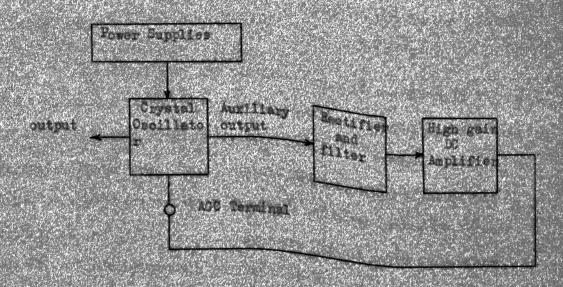
to sustain oscillations, but as the oscillations grow, the gain should be reduced to such an value that A > 1 only for the fundamental frequency. This definitely suggested going for a Automatic Gain Control (AGC).

3.3 Monitor Crystal Oscillator Design:

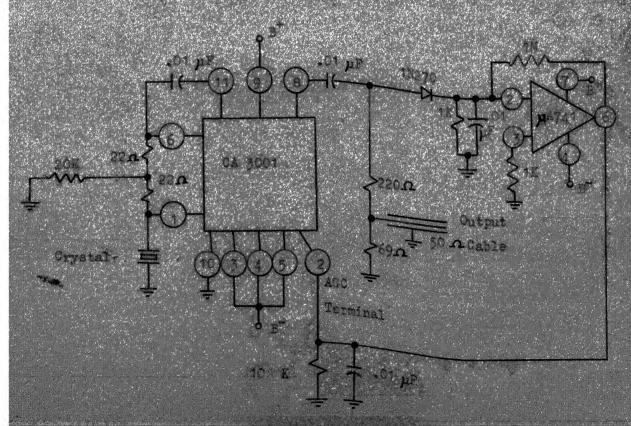
The scheme for the amplitude stabilized AGC oscillator is shown in Figure 4a. The integrated circuit (IC) amplifier to be used in the "Crystal Oscillator" Circuit must have following features,

- (1) Differential amplifier configuration, so that a positive feedback is obtained as in Figure 3f.
- (2) Emitter follower outputs, so that output impedence is low
- (3) AGC facility
- (4) Bandwidth larger than 6.5 MHz

The IC which fulfils all these requirements is the RCA type CA 3001 (for specifications refer to Appendix B). The final circuit using a 6.5 MHz AT-cut crystals is shown in figure 4b. A diode and a RC combination performs the rectification and filtering so that d-c voltage proportional to the amplitude of oscillation is obtained. The operational amplifier provides gain and inversion thus gives the desired negative output d-c voltage which is connected to the AGC is used terminal of CA 3001. The IC Fairchild type µa 741 for the operational



(E) CENOTIFE DESCRIC



Migure: 4 DETAIL OF OSCILLATOR CIRCLIA

amplifier. obviates the need for external frequency compensation and thus reduces the component count on the monitor oscillator Printed Circuit board.

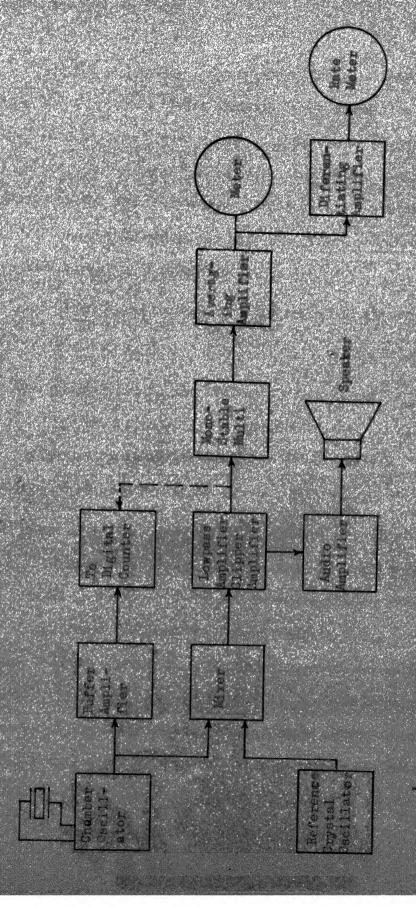
The details of the oscillator circuit are shown in Figure 4. This particular configuration has a number of advantages. Firstly, it eliminates mode jumping problem; secondly, it works for many crystals for the same class; thirdly, stabilization of amplitude leads to a good stability in frequency and output waveform becomes sinusoidal and lastly, use of integrated circuit makes it possible to put the oscillator-circuit very near to the monitor crystal thus reducing parasitic lead inductances and capacitance improving the frequency stability.

CHAPTER A

QUARTZ CRYSTAL MONITOR.

4.1 Scheme for measuring thickness and rate of deposition of thin films:

The sensor namely the quartz crystal oscillator put inside the vacuum chamber alongside the substrate, shows a reduction in frequency as the film gets deposited on the crystal. in frequency of is proportional to the mass of the film and hence its thickness. The scheme for measuring this change in frequency of is shown in Figure 5. There are two ways of doing this. One is to use an electronic counter to find of directly, Alternately a DC level proportional to Af is obtained which is measured using a voltmeter. Frequency to DC voltage conversion is achieved as follows. The chamber oscillator output is mixed with the reference crystal oscillator output. The mixer output is passed through a low pass filter and a clipper amplifier. The square wave output from the clipper amplifier is fed to a monostable multivibrator. Averaging the monostable output gives a DC level proportional to the difference frequency. A calibrated panel meter is used to indicate the thickness. The DC level proportional to the beat frequency is differentiated to indicate the rate of deposition. When the reference oscillator is tunable, an audic note indicating the change in frequency is obtained by the use of an audio amplifier and speaker. This is also used for rough monitoring.



Peurer 5 The Calaba medation languagement source

Since the analog indications on the panel meters cannot be read to an accuracy better than 2% of the fullscale, electronic counter is used when better accuracy in thickness or rate measurements is required. The counter can be made to count the chamber oscillator frequency or the beat frequency. When the beat frequency is counted, the reference oscillator should also be a crystal controlled to a compatible frequency stability.

Circuit realization for this scheme is discussed in the next section.

4.2 Circuit realization and design:

The aim of this work has been to design and fabricate the whole system using integrated circuits (ICs), since the use of ICs results in superior performance and reliability.

4.21 Mixer design:

Mixing is achieved using the variable gain property of a differential amplifier. The signal with frequency f_1 fed to the base of the current source transistor, changes the quiescent current through the differential amplifier configuration changing its gain. The other signal, whose frequency is f_2 , is applied between the differential input terminals. The modulated output is obtained at the differential output terminals which contains the sum and difference frequencies $f_1 + f_2$ and $f_1 - f_2$. The former is eliminated by a low-pass

filter to get the beat frequency (f1 - f2).

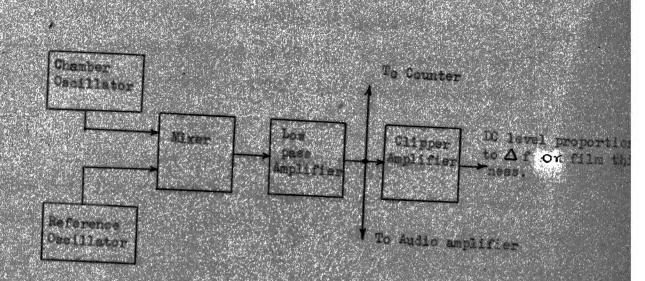
Thus, requirements for the IC differential amplifier to be chosen to perform the mixing are that it should have a Bandwidth much larger than f_1 or f_2 (6.5 MHz) and adequate gain for the beat frequency.

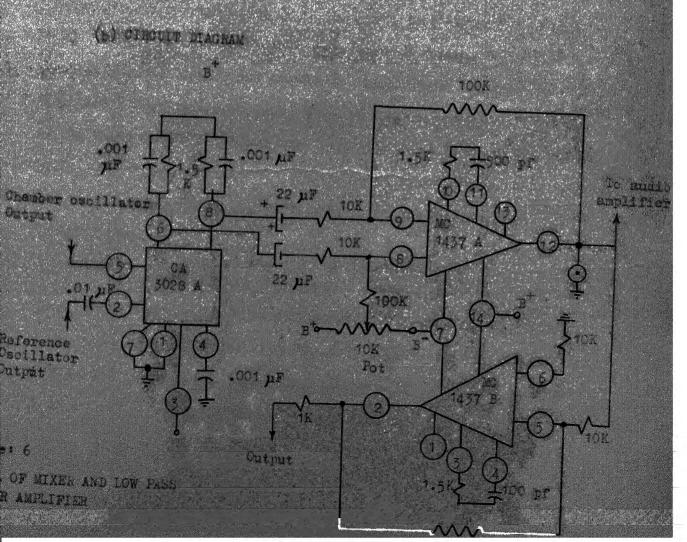
The IC RCA type CA 3028A (Refer to Appendix B for specifications) is chosen for this and low-pass RC filters are incorporated in the collector circuit. The lower cut-off frequency is kept at 100 KHz. The circuit diagram is shown in Figure 6b and its details in Appendix B. The amplitude of the beat frequency is found to be of the order of 100 mV.

4.22 Low-pass and clipper amplifier:

Function of this stage is to attenuate the unwanted high frequency to atleast 60 db below the beat frequency level. Thus, the low-pass amplifier and clipper amplifier, each provide an attenuation of 30 db at 13 MHz and provide large enough gain to give a square wave shape to the beat frequency. Since the mixer output is 0.1 V, and the clipping level about ± 5 volts (for ± 6 V supplies), the gain required is about 1000.

The IC dual operational amplifier Motorola type MC 1437 fulfils all these requirements in terms of gain, dynamic range and frequency response (refer to Appendix B for specifications).





A 100 K resistor and a 10 K potentiometer are used to provide the input-off-set null as shown in Figure 6b. The a-c coupling employed between the mixer and the low pass amplifier should have a lower cut off frequency below 100 Hz. Thus a capacitance of 22 µF is used for this purpose.

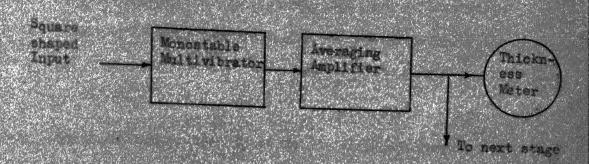
Input to the audio amplifier is given after the low pass amplifier, while the clipper amplifier output is fed to the monostable multivibrator.

4.23 Monostable Multivibrator:

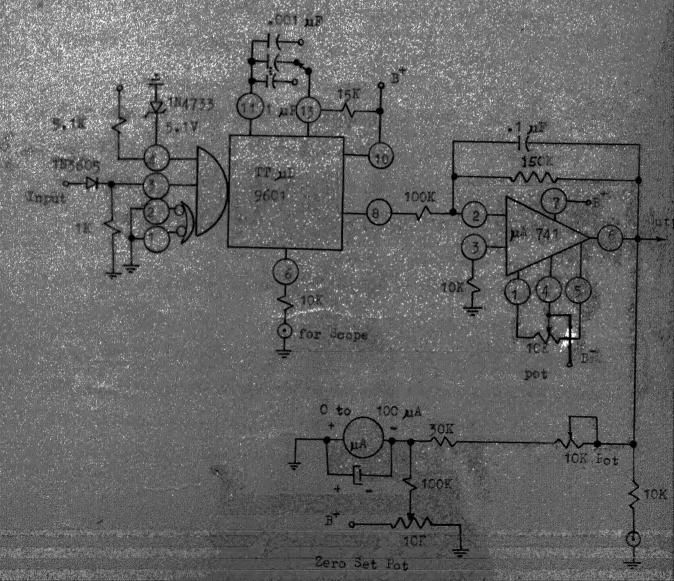
The monostable multivibrator provides a single standardised rectangular pulse output when it is triggered once. Since it is triggered by the beat frequency, the output when averaged gives a d-c voltage linearly related to the beat frequency. Thus, for a linearity better than 2% for the highest frequency (100 KHz) the rise and fall times of the pulse should be better than 0.1 pages, which is 1% of the minimum period (10 pages.). Further the amplitude and pulse width stability with respect to power supply voltage variation is an important consideration.

The Fairchild type IC TPuL 9601 meets requirements mentioned above (refer to Appendix B for specifications). Input to pin 3 is given through a diode to block the negative excursions. Pin 4 is kept at + 4.5 volts by a zener diode as required by the device (Fig. 7b) In this multivibrator the output pulse width is controlled by the time

a) BLCCK DIAGRAU FOR MONOSTABLE MILITUREARCH AND AVERAGING



(B) CONTRACTOR MARKIN



Ruser 7 DEPART OF MONOSTABLE MULTIVISHATOR AND AVERAGING AMPLIFUER

constant of a resistor - capacitor combination to be provided externally. This time constant is altered in order to obtain full scale deflection for the highest frequency measured in various frequency ranges. The range selection is obtained by switching the capacitances. The pulse width at any given frequency range is decided as given below.

The capacitances are chosen for the different ranges (30 KHz, 10 KHz, 3 KHz, 1 KHz, 300 Hz and 100 Hz) such that pulse width remains at half the period corresponding to the highest frequency in that range.

An auxiliary d-c coupled output (pin 6) is available for monitoring the pulses on an oscilloscope. A 10 K resistor is provided to protect the IC against accidental short circuit of the monitoring terminal.

4.24 Averaging amplifier:

A Fairchild type AA 741 internally compensated high gain amplifier is used for averaging the output of the monostable multivibrator (specifications of AA 741 are given in Appendix B). The lower cut off frequency of the amplifier is kept at 10 Hz so that the d-c level at the output can change with a time constant of 1/60th sec. To avoid very large capacitance a 150 K resistance is chosen at the feedback element with a 0.1 AF capacitor across it. The gain

of the amplifier is arranged such that it does not saturate for the highest frequency measured in any given frequency range. The output voltage corresponding to this situation is kept at 4 volts. Since the saturation output level of the amplifier is 5 volts, the voltmeter used to measure the output voltage does not need any protection (only 25% overloading).

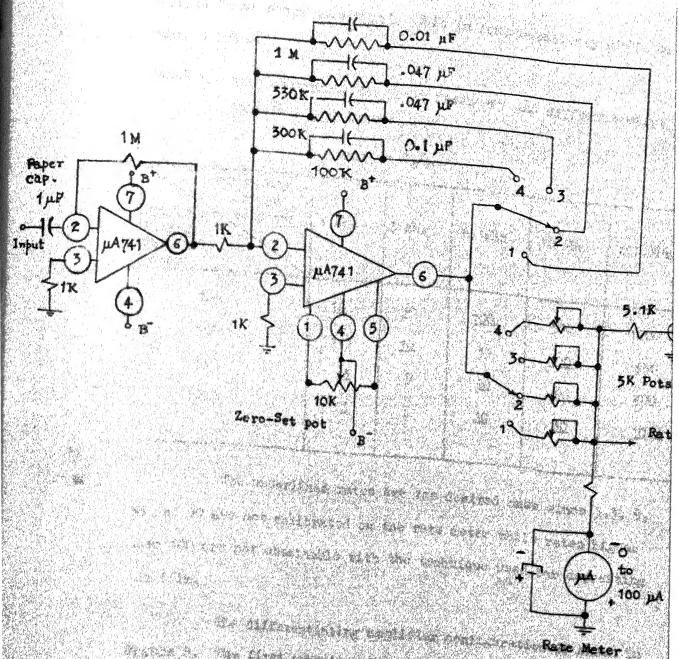
4.25 Differentiating or rate amplifier:

The information about the rate of deposition is obtained by differentiating the output voltage which varies in accordance with the thickness. The calibration of the rate meter depends upon the range selected by the "thickness range switch". Thus, in order to obtain full scale deflection on the rate meter for a given rate of deposition, the gain of differentiating amplifier must be adjusted.

Given a thickness range and a rate for which full scale deflection on the rate meter is desired, the gain of the differentiating amplifier should be

$$\frac{\mathbf{thickness} \text{ range } \underline{\mathbf{in}} \ \Lambda}{\mathbf{rate} \ \mathbf{in} \ \Lambda} \quad \mathbf{eqn} \quad (7)$$

In equation (7), H_Z can be used interchangeably with A since they are directly proportional to each other and appear in both the numerator and denominator. Thus for frequency ranges of 1 KHz, 3 KHz, 10 KHz, 30 KHz, 100 KHz and rate ranges of 1,3,10,30 and 100 Hz/sec, we find that gains of 100, 300, 350 and 1000 are to be provided for



DIFFERENTIATING AMPLIFIER AND RAFE METER

Pigure : 8

desired "rate range selection". This is comprehensively given in

ABLE 1 Rate obtainable for given frequency and differentiating amplifier gain ranges

Rate		in	Hz/sec	
------	--	----	--------	--

Frequency range selector setting		3 KHz	10 KH2	30 KHz	100 KHz
gain sel- ctor		Merchanists Commencer			
100	10	<u>30</u>	1 0 0	300	1000
300	3.3	10	33	<u>100</u>	<i>3</i> 30
330	3	9	30	90	3 0 0
1000	1	5	10	30	100
					- BO -

The underlined rates are the desired ones since 3.3, 9, 33 and 90 are not calibrated on the rate meter while rates higher than 100 are not obtainable with the technique used for depositing the film.

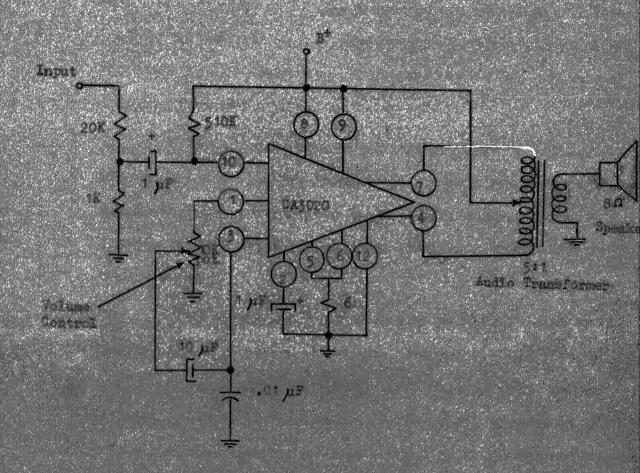
The differentiating amplifier configuration is shown in Agure 8. The first operational amplifier performs the differentiation while other gives the extra amplification desired. The differential amplifier gain is given by product RC which we want to be of the order of 100 to 1000. Therefore, R must range in several Mos while C in

in several µF. As we go for electrolytic or tantalum capacitors the leakage current itself can put the amplifier in saturation. Therefore it is imperative to use paper capacitor to reduce the leakage current. Thus the limiting values chosen are 1 µF for paper capacitor and 1 Mn for feedback resistor. This gives us a gain of only 1. An cascaded amplifier providing a variable gain of 100, 300, 330 or 1000 for d-c voltage fulfils the requirement for the differentiating amplifier gain. Transient changes in the voltage proportional to thickness are avoided in the rate indication by making the amplifier response slow by providing RC time constant of the order .01 sec in the feedback.

4.26 Audio amplifier

The RCA type CA 5020 integrated circuit is used for the audio amplifier (for specifications refer to Appendix B). The input to the audio amplifier is the difference frequency output voltage from the lowpass amplifier stage (Figure 6b). A potential divider is used so that input to the CA 3020 is about 100 mV (Figure 9). With 1 μ F capacitance in the input circuit, the lower cut off frequency is about 3 Hz (R_{in} 10 \geq 50 K). The upper cut-off frequency is decided by the capacitance connected between pin 3 and ground. The upper cut off frequency for .01 μ F capacitance connected between pin 3 and ground is 16 KHz (R_{in} 3 \leq 1K). Volume control is provided by the 10 K potentiometer.

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4.3 Calibration:

It is very convenient to calibrate the thickness and rate meters in terms of frequency (Hz) and rate of change of frequency (Hz/Sec) respectively as the thickness and rate of deposition of the films have linear relationship with change in frequency and rate of change of frequency.

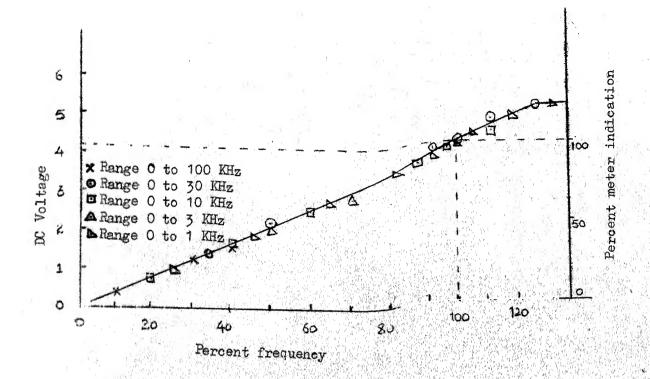
The thickness meter is calibrated to give a full scale deflection for the highest frequency in any given range. This is done by adjusting the pulse width of the monostable multivibrator (by choosing proper C_{χ}) until a full scale reading is obtained on the thickness meter.

The rate meter is calibrated by feeding in a ramp of the desired slope (in volts/sec) and then adjusting the gain for ramp such that a full scale deflection is obtained. This is clear from the calibration curves given in figure 11. The d-c level corresponding to the full scale reading are kept slightly below the saturation levels of JA741 so that both linear response and protection of the meter are achieved simultaneously.

4.4 Monitor Operation:

The sensor head is placed alongside the substrate in such a way that the substrate and monitor crystal are at the same level from the evaporation source. Proper ranges for thickness and rate

are selected. After evacuating the chamber to a pressure less than 10^{-5} torr, evaporation is started and the rate is adjusted by controlling the current through the filament. The thickness meter indication is made zero with the help of "Zero-set" control. At this instant, the shutter covering the substrate is removed and deposition of film on the substrate is started. The shutter is replaced again as soon as the desired thickness is indicated on the meter. Then the evaporatio source power is gradually decreased to zero.



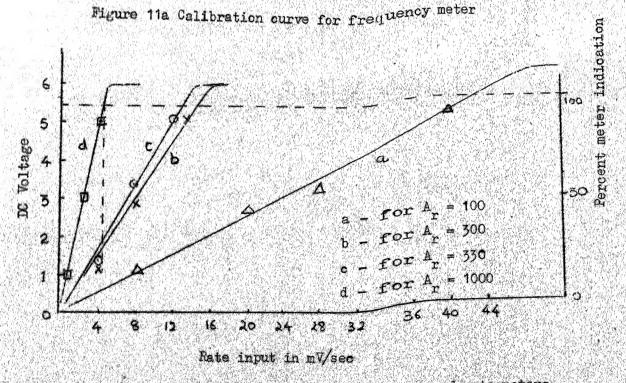
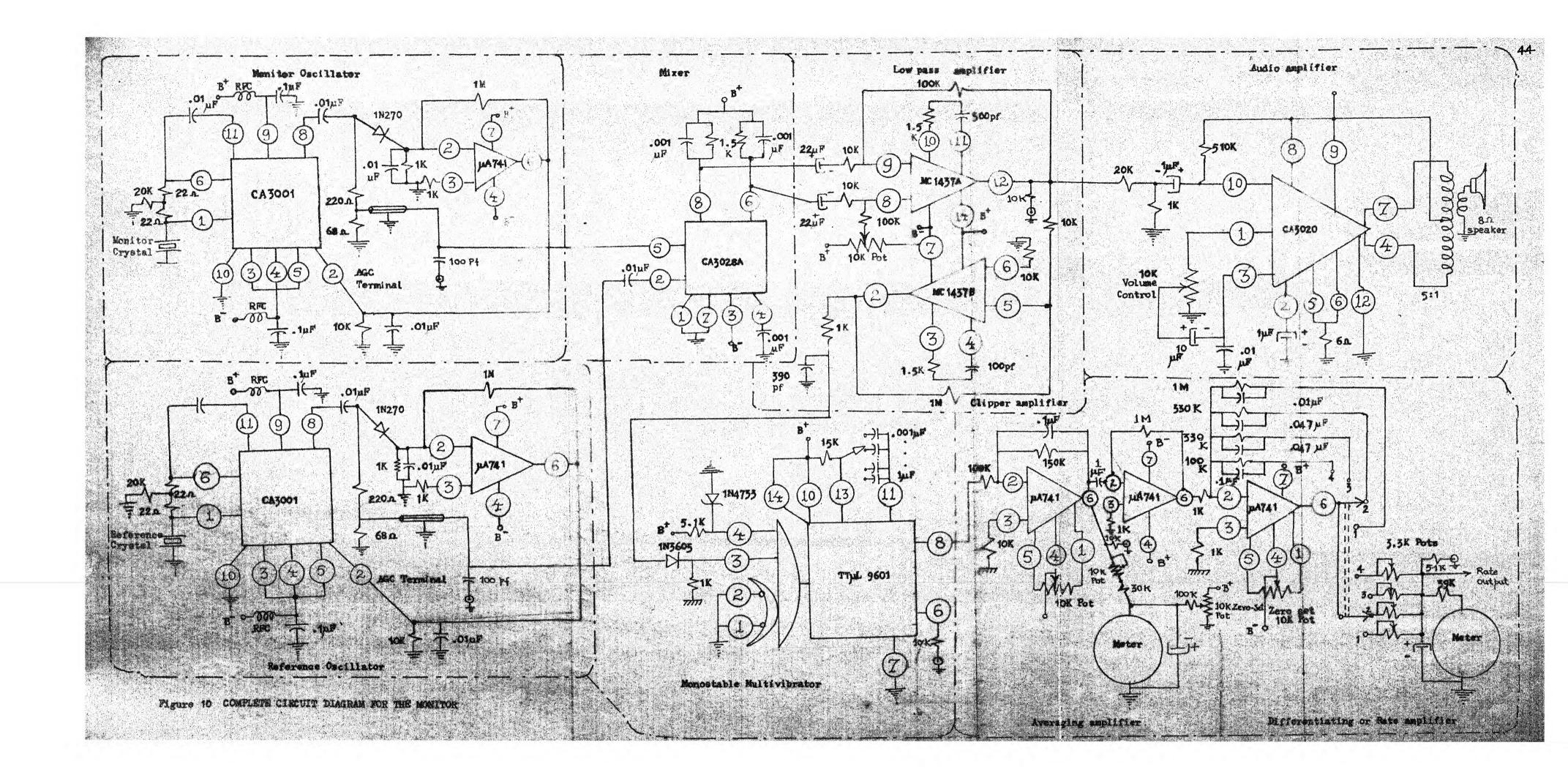


Figure 11 Calibration curves for frequency and rate meters



CHAPTER 5

EXPERIMENTAL OBSERVATIONS AND RESULTS

5.1 Monitor crystal oscillator circuit;

Experiments were performed on many crystals without using AGC and it was found that a certain amount of overdrive (AB71) is required to cause sustained oscillations with a number of different crystals. Figure 12a shows the effect of overdrive. The waveform is more like a square wave and hence has lot of harmonic content. With the same overdrive some crystals oscillate at overtone frequencies while others jump to an overtone frequency when film gets deposited on the crystal. The wave form corresponding to an overtone frequency, is shown in Figure 12b. When AGC is used, a large number of crystals of the same class, either unplated or plated (loaded with film material), continue to oscillate in their fundamental mode. The harmonics are eliminated and waveform is sinusoidal as shown in Figure 12C. The amplitude is also stabilized resulting in a better frequency stability. For the crystal oscillator made, the frequency gets stabilized within a Hiz after about 5 minutes from the time when the power is switched on.

5.2 Calibration of the monitor for copper and silver:

Copper was deposited simultaneously on the monitor crystal and an auxiliary substrate. The change in the crystal oscillator frequency was observed. The thickness of the copper film was measured

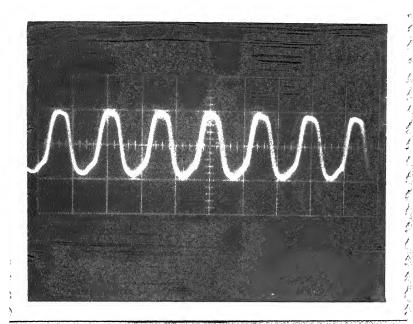


Figure 12a
Oscillator output
without AGC

Time scale: 0.1 sec/cm Voltage scale: 50 mv/cm

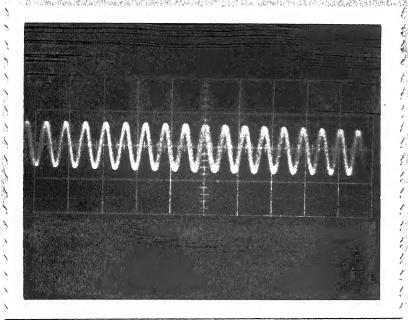
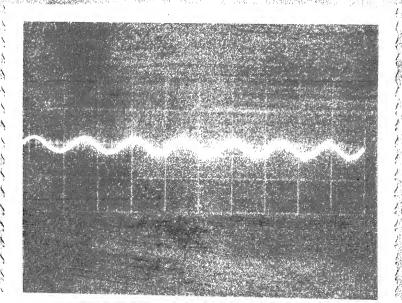
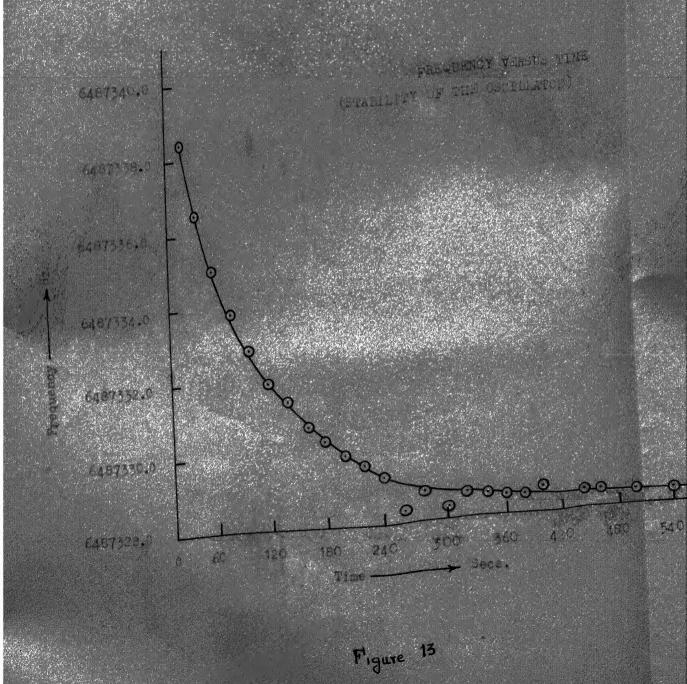


Figure 12b
Oscillations at
Jrd harmonic because
of either excessive
everload or film
deposition

Figure 12c
Oscillator output
with AGC





by the optical interferometric method as described in Appendix A.

Experiment is repeated again and change in frequency $\triangle f$ and thickness of the film are determined. The calibration curve obtained is shown in Figure 14a. The curve shows the linear relationship between the change in frequency and the thickness.

The procedure was repeated for silver as the film material. The calibration curve for silver isoshown in Figure 6146. These curves are used to read the thickness of films of these materials when the change in frequency \triangle f is measured.

For a typical run following are the observations and results:

Pressure in the evaporation chamber = 10⁻⁵ terr

Current through the filament = 22 Amps.

Monitor crystal oscillator frequency = 6536.3 KHz at the time when deposition starts

Frequency at the end of deposition = 6523.0 KHz

Frequency of the reference oscillator = 6530.0 KHz

Frequency range selected for thickness) = 0 to 30 KHz meter

Frequency indicated on panel at the start of deposition

Frequency indicated at the end) = 20.5 KHz of deposition

Differential amplifier gain selected = 300

Rate indicated by meter

Time of deposition

Area of the monitor crystal exposed to the evaporant

Area of the crystal

≛ 10 Hz/sec.

= 25 min.

= .28 sq. cm.

1.8 sq. cm .

Results:

Frequency shift

Thickness of the film as measured by MBI

Total mass deposited

Minimum detectable mass

Frequency change per A for copper

= 13.3 KHz

3000 ± 80 A

≟ 8 x 10⁻⁵ gm

= 10⁻⁹ gm/cm²

) = 4.4 Hz/A

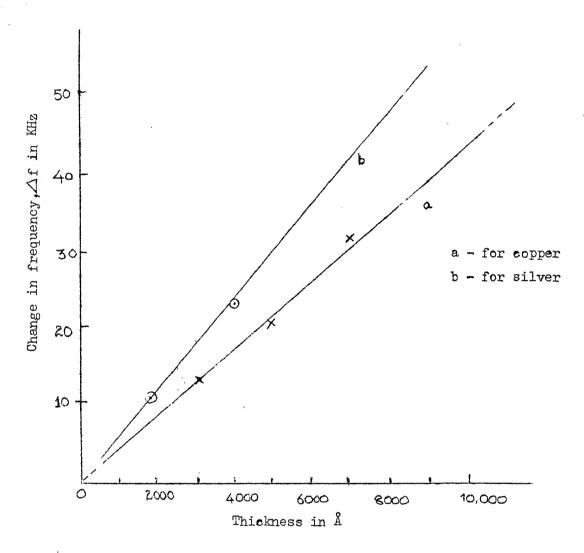


Figure 14 Calibration of the monitor for copper and silver

CHAPTER 6

CONCLUSIONS

A quartz crystal monitor for measuring thickness and rate of deposition of thin films has been designed and fabricated. A particular problem, namely the mode jumping problem, has been eliminated by employing AGC scheme in the monitor crystal oscillator circuit. Employing AGC also leads to a stability in frequency better than 1Hz/hour. The use of linear integrated circuits has minimised the component count and thus have resulted in a compact water cooled sensor-head. The whole system has been made using ICs in view of superiority in performance and reliability.

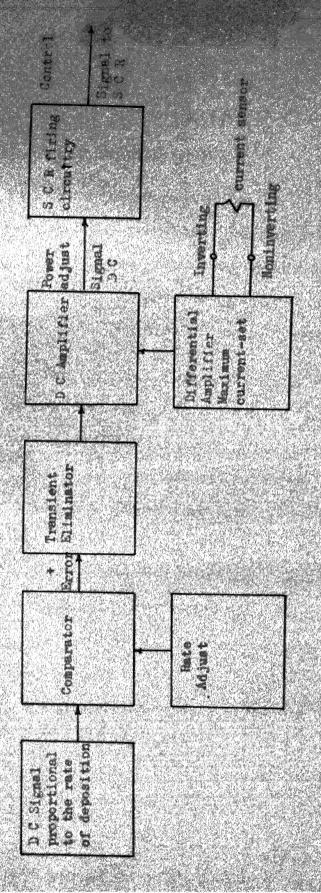
Experimental results show that mass sensitivity of the order of 10^{-9} gm/cm² can be achieved.

CHAPTER 7

FUTURE WORK

In-situ measurement of thickness and rate of deposition is made by the quartz crystal monitor fabricated. The monitor can be used with a compatible "automatic rate control unit" (shown in Figure 15) to control and stabilize the rate of deposition. The d-c signal proportional to the rate of deposition is compared with the reference rate-adjust d-c level and the error signal after amplification is made to control the friggering of the SCRs which controls the evaporation source power.

Automatic control of thickness can be achieved by providing a pre-set thickness pointer in the thickness meter and when thickness indications becomes equal to the present value, a relay can be made to operate the shutter or cut-off power supply to the evaporation source.



RIGGE DISCREM FOR THE ATTURNETO RATE CONTROL UNIT

Appendix A

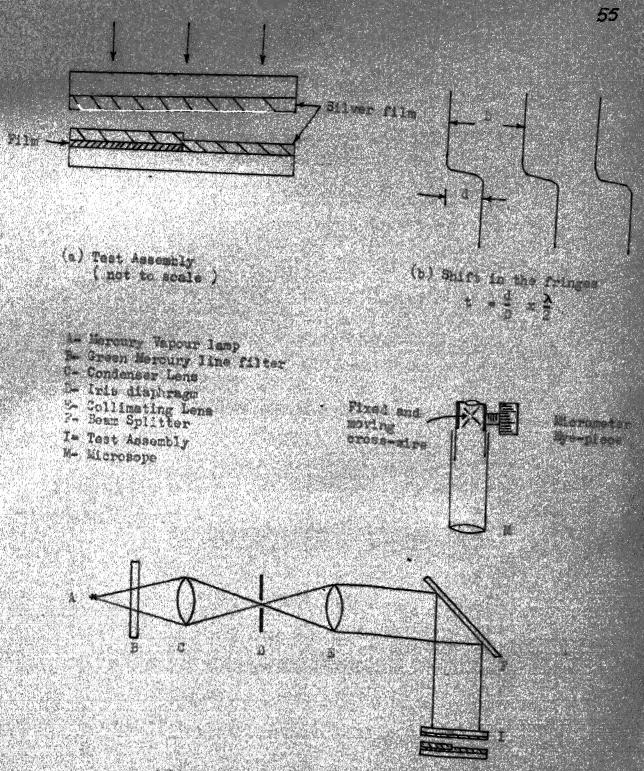
Determination of thickness of thin films by Multiple-Beam-Interferometer:

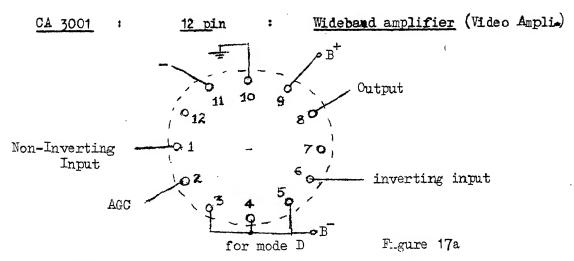
Primary measurement of thickness of thin films has been by done Multiple Beam Interference. The technique of thickness measurement of thin films by Multiple Beam Interference is due to Tolansky. (1948, 1955, 1960). Optimization of the Tolansky technique is discussed by Flint (1967). The method is described briefly below.

Thin film AB, in Figure 16a whose thickness is required is deposited on a flat surface to cover only a part of the surface. Over this is deposited by evaporation, a film of silver 700 Å thick. It has been well established that such deposition contours the underlying surface accurately. There is now a step in height separating FQ and QR which is the same height as that of the film AB. This composite surface is now matched, slightly filted, against an optical flat, half silvered or with a (high reflectivity, low absorption) multilayer dielectric coating. This assembly is illuminated with parallel monochromatic light, from above, so that multiple beam reflection wedge-fringes are secured. The step appears in fringes and can be measured accurately. The observed pattern of fringes is shown in figure 16b. If the fringe separation is D, the step in fringes is d and the wavelength of the monochromatic light is λ (in λ), then

't_f', thickness of the film $= \frac{d}{D} \times \frac{\lambda}{2} \dots \lambda$

The schematic for this method is given in figure 16c.





Features:

- . for use in video and comm. equipment
- . Balanced Diff. Amp with controlled constant current
- . Emitter follower input & output
- . optional 15 pt output coupling cap.

<u>Applications</u>

- . Schmitt Trigger
- . DC, IF video amplifier

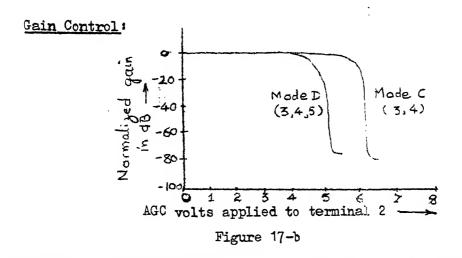
- . Mixer
- . Modulator

Highlights AGC range ... 60 dB typ. Bandwidth ... 16 MHz Input resistance ... 50 appr. Output resistance ... 70 Voltage gain ... 19 dB typical

. Input offset voltage . . 1.5 mV typical

Electrical Characteristics:

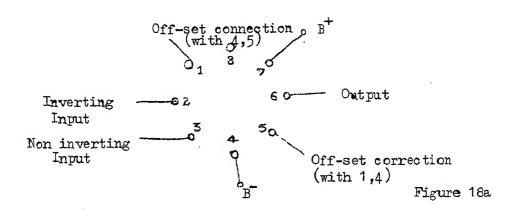
Operation Mode	1		supply	Power Dissi- pation	Single ended voltage gain at 1MHz
D(3,4,5)	<u>+</u> 6 v	8.7 mA	-5•5mA	85 mW	16.4 dB



Fairchild Type A741

Internally compensated operational amplifier

Connections



Electrical Characteristics:

 $Vs = \pm 15 V$

Typical

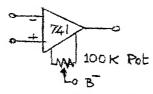
Input resistance

1 M

Large signal voltage gain

Output swing $R_{T} \geqslant 2 \text{ K}\Omega$

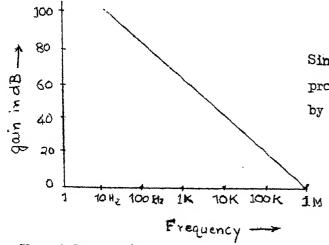
10 V



Voltage offset Null Circuit

Figure 18b

Open Loop gain - Vs - frequency



Since no compansation is provided cut off is decided by gain

Figure 18c

Closed loop gain

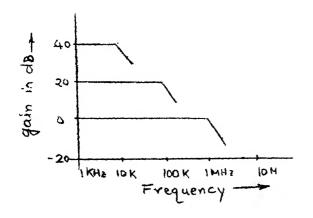
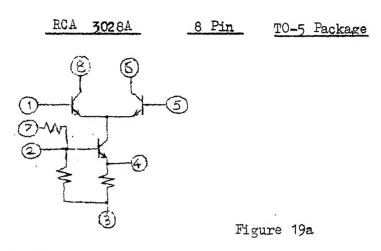


Figure 18d

Therefore A741 is suitable for low-frequency amplification with gain as a function of feedback elements.



Features: (1) Use in comm. and industry

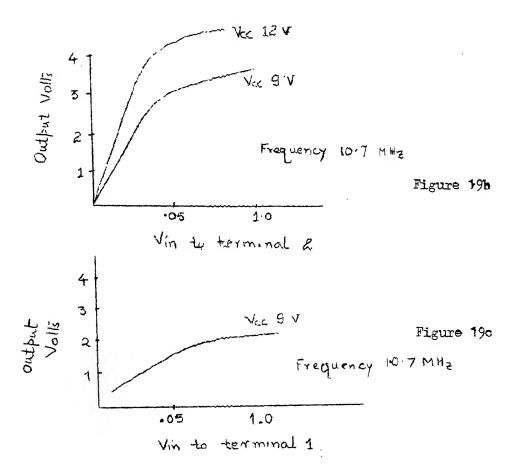
- (2) Balanced diff. ampl. with constant current source
- (3) Single ended operation only one power supply needed
- (4) Operation from DC to 120 MHz
- (5) Balanced AGC capability
- (6) Wide operation current range

Applications: (1) RF and IF amplifiers

- (2) Converter in the commercial FM band
- (3) Oscillator
- (4) Mixer
- (5) Limiter
- (6) DC amplifier

for +9 volt supply

Characteristic	Terminal	[ypical
Quiscent current	I6 or I8	2.5
Input admittance		≈t 6.5 MH ,5 + j.4 x 15 ³
Voltage gain	Load 1 K freq. 10.7 MH	32



MC 1437 Dual Operational amplifiers

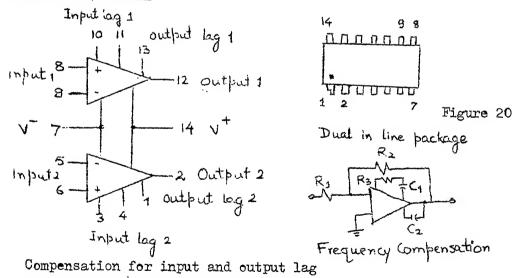
Designed to use as summing amplifiers, integrators, or amplifiers with operating characteristics as a function external feedback components.

Features:

- High performance 45,000 open loop gain

output impedence 30

Equivalent Circuit:



	gain	eut-off frea	R	R ₂	R_{3}	C ₁	Co
I	10	300 KHz	10K	100K	1.5K	500pf	20pf
I	100	300 KHz	10K	1M	1.5K	100pf	3pf
Ī	1000	200 KHz	1K	1M	0	10pf	3pf

Retriggerable Monostable Multivibrator 9601 TT \(\mu \)L

A Fairchild Compatible Current Sinking Logic Product

General description:

The TT L 9601 is a DC level sensitive retriggerable monostable multivibrator which provides an output pulse whose duration and accuracy is a function of external timing components only. The 9601 has excellent immunity to noise on the V_{CC} and ground lines. The 9601 uses TT L for high speed and high fan-out capability and is compatible with all devices in the CCSL family of IC's.

Features:

- (a) 50 ns to ∞ output pulse width range
- (b) Retriggerable 0 to 100% duty cycle
- (c) Complementary D.C. level sensitive inputs.
- (d) Complementary outputs
- (e) CCSL compatible
- (f) Optional retrigger lock-out capability
- (g) Pulse width compensated for V and temperature variations

Absolute Maximum Ratings:

V Pin Potential to Ground -0.5 to + 8.0 V

Input voltage D.C. -0.5 to + 5.5 V

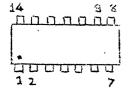
Input current -30 mA to + 5.0 mA

Voltage applied to output when output is high OV to + V oc value

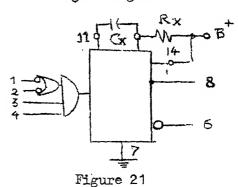
Current into output when output is low

50 mA

Dual-in-line package



Logic Diagram



Operational Rules

- (1) Range of R 10K to 30K
- (2) $C_{\mathbf{x}}$ may vary from 0 to any value necessary and obtainable
- (3) Output pulse width T,

T = 0.32
$$R_x$$
 C_x (1+ $\frac{0.7}{R_x}$)...(for C_x greater than 10³ pf)
Where R_x is in $K \cap C_x$ is in PF
T is in nS

Electrical Characteristics:

Quiescent Power Supply Drain Typical 25 mA ($V_{cc} = 5.5 \text{ V}$)

		•
CA 3020	Multipurpose Wideband Power Amplifier	
Features	. High power output class B amplifier	0.5 watt
	. Wide frequency range upto	8 MHz
	. High power gain	•
	. Single power supply for class B) operation with transformer	3 to 9 ♥
Absolute 1	Maximum ratings	
	Dissipation at 25°C	1 W
•	I _{4.5.6.7}	500 mA

Electrical characteristics

Total current drain (at	$v_{cc} = 9V) \dots$	21.5 mA
$P_{\text{omax}} (V_{\text{ce}} = +6V)$		300 m₩
Input resistance Ring		1000 typical

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